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(1,10-Phenanthroline-κ^2N,N')bis(2,2,6,6-tetramethylheptane-3,5-dionato-κ^2O,O')-nickel(II)

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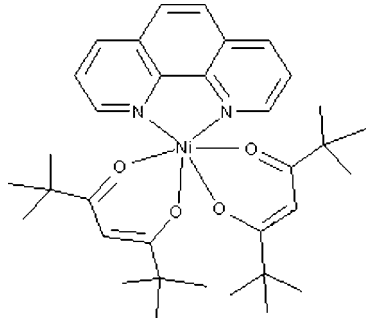
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.045; wR factor = 0.131; data-to-parameter ratio = 17.2.

The title compound, $[Ni(C_{11}H_{19}O_2)_2(C_{12}H_8N_2)]$, was obtained from the reaction of bis(2,2,6,6-tetramethylheptane-3,5-dionato)nickel(II), [Ni(dpm)], and 1,10-phenanthroline (phen). The Ni^{II} ion is coordinated by four O atoms from two dpm ligands and two N atoms from a phen ligand in a slightly distorted octahedral environment. The methyl C atoms of two of the *tert*-butyl groups are disordered over two sites, having approximate occupancies of 0.85 and 0.15 for the two components. In the crystal structure, there are no direction-specific interactions. Thermal studies showed that the title complex is stable to 623 K.

Related literature

For information on the synthetic procedure, see: Meštrović & Kaitner (2006). For information regarding the application of metal complexes with β-diketones, see: Soldatov *et al.* (1999, 2001, 2002, 2003); Soldatov & Ripmeester (2001*a*,*b*). For similar metal(II) (β-diketonates)$_2$ as well as for the properties of neutral molecules which form different types of supramolecular assemblies, see: Bučar & Meštrović (2003); Meštrović *et al.* (2004); Meštrović & Kaitner (2006). For the crystal and molecular structure of bis(2,2,6,6-tetramethylheptane-3,5-dionato)nickel(II), see: Cotton & Wise (1965). For the crystal and molecular structure of bis(acetylacetonato)-1,10-phenanthroline-nickel(II), see: Steblyanko *et al.* (1992).

For related literature, see: Allen (2002); Kaitner & Meštrović (1993).



Experimental

Crystal data

$[Ni(C_{11}H_{19}O_2)_2(C_{12}H_8N_2)]$
M_r = 605.44
Triclinic, $P\overline{1}$
a = 10.054 (2) Å
b = 10.386 (3) Å
c = 16.717 (2) Å
α = 89.69 (2)°
β = 83.48 (2)°
γ = 79.63 (3)°
V = 1705.8 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 293 (2) K
0.60 × 0.60 × 0.30 mm

Data collection

Philips PW1100 diffractometer with Stoe upgrade
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.65, T_{max} = 0.83
7711 measured reflections
7451 independent reflections
5139 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
3 standard reflections
frequency: 90 min
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.131
S = 1.02
7451 reflections
432 parameters
69 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.64 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni—O12	2.0110 (18)	Ni—O22	2.0320 (18)
Ni—O21	2.0176 (18)	Ni—N32	2.089 (2)
Ni—O11	2.0304 (18)	Ni—N31	2.094 (2)

Data collection: *STADI4* (Stoe & Cie, 1994); cell refinement: *X-RED* (Stoe & Cie, 1994); data reduction: *X-RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2569).

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supplementary materials

Acta Cryst. (2008). E**64**, m230-m231 [**doi:10.1107/S160053680706672X**]

(1,10-Phenanthroline-κ^2N,N')bis(2,2,6,6-tetramethylheptane-3,5-dionato-κ^2O,O')nickel(II)

B. Kaitner, K. Mesarek and E. Mestrovic

Comment

As part of our research of the study of metal β-diketonates complexes we have prepared the title compound by reaction of bis(2,2,6,6-tetramethyl-3,5-heptanedionato-κ^2 O,*O*') nickel(II) with 1,10 phenantroline (Bučar and Meštrović, 2003, Meštrović *et al.*, 2004, Meštrović and Kaitner 2006). After the work by Soldatov and his group this material was recognized as a smart sorbent and as a functional organic zeolite analogue (Soldatov *et al.*, 1999, 2001, 2002, 2003; Soldatov & Ripmeester, 2001*a*,b).

Another very important field of application is based on the thermal stability and volatility of metal β-dionates which makes them important precursors in production of high temperature supraconductors using technique of metal organic chemical vapour deposition (MOCVD). Based on molecular structural properties of metal(II)(β-diketonates) $_2$ as well as on properties of neutral molecules we can obtain different properties of materials. Necesssary prerequisites of source precursors for any MOCVD proces are thermal stability, sufficient and stable evaporation, and good delivery properties under process conditions. The success of a potential compound, mostly depends on the properties of molecular precursors, since their nature and architecture both make the quality of materials. Nevertheless, relationships between the precursor molecular architectures and their properties still remain a challenge in the area of materials science.

The reason for using 2,2,6,6-tetramethyl-3,5-heptanedion (dipivaoilmethan, Hdpm) was the non-polar property of *tert*-butyl groups in the title molecule giving no possibility for interaction between molecules of complex. This fact is very important in preparation of material for metal organic chemical vapor deposition. We introduced 1,10 phenantroline as aditional part for achiving thermal stability of the substance.

We obtained the adduct molecule through reaction of bis(dipivaloimethan)nickel(II) with 1,10-phenantroline. The Ni^{II} ion is in a slightly distorted octahedral environment formed by two dipivaloilmethanate ligands and one 1, 10-phenantroline ligand. The Ni—O bond distances range from 2.012 (2) Å to 2.033 (2) Å and are longer than the bond distances found in the free Ni(dpm)$_2$ complex (Cotton & Wise, 1965) which range from 1.839 Å to 1.844 Å. All other bond distances are similar to all other compounds in this class (Allen, 2002).

Experimental

Bis(2,2,6,6-tetramethyl-3,5-heptanedionato-κ^2 O,*O*') (1,10-phenanthroline-κ^2N,N')nickel(II) was prepared by the published method (Meštrović and Kaitner, 2006). 1 mmol (190 mg) of phenantroline was disoved in 10 ml of acetone. 1 mmol (415 mg) of bis(2,2,6,6-tetramethyl-3,5-heptanedionato-κ^2 O,*O*') nickel(II) was added to a warm solution of phenantroline. Green crystals were obtained overnight. The crystal suitable for single-crystal *X*– ray diffraction was obtained by evaporation of diluted acetone solution of the title compound over two weeks.

Refinement

H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distance from 0.93 to 0.96 Å. They were treated as riding atoms, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C) for methyl C atoms. The methyl groups on atoms C18 and C24 are disordered over two sites with the ratio of the refined occupancies being 0.853 (7):0.147 (7) and 0.846 (7): 0.154 (7), respectively.

Figures

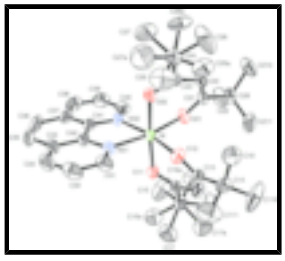


Fig. 1. The molecular structure of the title complex showing the numbering sheme and displacement ellipsoids drawn at the 30% probability level. The H atoms have been omitted for clarity.

(1,10-Phenanthroline-κ^2*N*,*N*')bis(2,2,6,6-tetramethylheptane- 3,5-dionato-κ^2*O*,*O*')nickel(II)

Crystal data

$[Ni(C_{11}H_{19}O_2)_2(C_{12}H_8N_2)]$	$Z = 2$
$M_r = 605.44$	$F_{000} = 648$
Triclinic, $P\overline{1}$	$D_x = 1.179$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.054$ (2) Å	Cell parameters from 25 reflections
$b = 10.386$ (3) Å	$\theta = 10$–15^o
$c = 16.717$ (2) Å	$\mu = 0.61$ mm^{-1}
$\alpha = 89.69$ (2)°	$T = 293$ (2) K
$\beta = 83.48$ (2)°	Prism, green
$\gamma = 79.63$ (3)°	0.60 × 0.60 × 0.30 mm
$V = 1705.8$ (7) Å^3	

Data collection

Philips Stoe upgrade diffractometer	$R_{int} = 0.033$
Radiation source: fine-focus sealed tube	$\theta_{max} = 27.0^o$
Monochromator: graphite	$\theta_{min} = 2.1^o$
$T = 293(2)$ K	$h = -12 \rightarrow 12$
ω scans	$k = -13 \rightarrow 13$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 21$
$T_{min} = 0.65$, $T_{max} = 0.83$	3 standard reflections

7711 measured reflections	every 90 min
7451 independent reflections	intensity decay: 1%
5139 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.131$	$w = 1/[\sigma^2(F_o^2) + (0.0711P)^2 + 0.3734P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
7451 reflections	$\Delta\rho_{max} = 0.64$ e Å^{-3}
432 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
69 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger. Disorder was refined with restraints on bond distances of both orientations and on ADPs of atoms of the minor orientation. Occupancie of minor orientations of both t-buthyl residues refined to 0.85

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Ni	0.32575 (3)	0.31322 (3)	0.188562 (17)	0.04550 (12)	
O11	0.47578 (17)	0.18444 (17)	0.23263 (10)	0.0548 (4)	
O12	0.19611 (17)	0.27128 (19)	0.28160 (10)	0.0608 (5)	
O21	0.38533 (17)	0.45956 (17)	0.24617 (10)	0.0576 (4)	
O22	0.17421 (17)	0.44893 (17)	0.15129 (10)	0.0545 (4)	
N31	0.2711 (2)	0.1796 (2)	0.11057 (13)	0.0560 (5)	
N32	0.4651 (2)	0.3236 (2)	0.08706 (12)	0.0525 (5)	
C11	0.4787 (2)	0.1676 (2)	0.30770 (14)	0.0493 (6)	
C12	0.3645 (3)	0.1894 (3)	0.36497 (16)	0.0616 (7)	
H12	0.3790	0.1724	0.4183	0.074*	
C13	0.2307 (3)	0.2343 (3)	0.34972 (16)	0.0558 (6)	
C14	0.1124 (3)	0.2384 (3)	0.41701 (18)	0.0710 (8)	

C19	−0.0004 (4)	0.3504 (4)	0.4040 (3)	0.1095 (15)	
H19A	−0.0309	0.3401	0.3524	0.164*	
H19B	0.0325	0.4314	0.4058	0.164*	
H19C	−0.0749	0.3511	0.4455	0.164*	
C110	0.1533 (5)	0.2546 (7)	0.5014 (2)	0.161 (3)	
H11A	0.1994	0.3278	0.5022	0.241*	
H11B	0.2129	0.1766	0.5150	0.241*	
H11C	0.0734	0.2696	0.5398	0.241*	
C111	0.0592 (5)	0.1104 (4)	0.4104 (3)	0.136 (2)	
H11D	−0.0230	0.1141	0.4464	0.204*	
H11E	0.1264	0.0386	0.4247	0.204*	
H11F	0.0408	0.0981	0.3562	0.204*	
C18	0.6206 (3)	0.1182 (3)	0.33414 (16)	0.0650 (7)	
C15	0.7313 (4)	0.1426 (8)	0.2697 (3)	0.133 (3)	0.853 (7)
H15A	0.7289	0.2351	0.2648	0.200*	0.853 (7)
H15B	0.7168	0.1069	0.2192	0.200*	0.853 (7)
H15C	0.8184	0.1013	0.2842	0.200*	0.853 (7)
C16	0.6370 (5)	0.1701 (8)	0.4154 (3)	0.123 (3)	0.853 (7)
H16A	0.7239	0.1300	0.4308	0.184*	0.853 (7)
H16B	0.5659	0.1502	0.4542	0.184*	0.853 (7)
H16C	0.6318	0.2632	0.4130	0.184*	0.853 (7)
C17	0.6373 (6)	−0.0320 (4)	0.3389 (4)	0.118 (2)	0.853 (7)
H17A	0.7277	−0.0678	0.3507	0.176*	0.853 (7)
H17B	0.6224	−0.0668	0.2882	0.176*	0.853 (7)
H17C	0.5723	−0.0548	0.3806	0.176*	0.853 (7)
C15A	0.704 (2)	0.008 (2)	0.2823 (16)	0.091 (8)	0.147 (7)
H15D	0.7168	0.0361	0.2276	0.136*	0.147 (7)
H15E	0.6584	−0.0657	0.2847	0.136*	0.147 (7)
H15F	0.7917	−0.0174	0.3016	0.136*	0.147 (7)
C16A	0.675 (2)	0.2465 (13)	0.338 (2)	0.092 (9)	0.147 (7)
H16D	0.6111	0.3086	0.3717	0.138*	0.147 (7)
H16E	0.6865	0.2809	0.2845	0.138*	0.147 (7)
H16F	0.7607	0.2303	0.3593	0.138*	0.147 (7)
C17A	0.616 (4)	0.066 (4)	0.4195 (11)	0.125 (12)	0.147 (7)
H17D	0.5637	0.1319	0.4562	0.187*	0.147 (7)
H17E	0.7064	0.0427	0.4341	0.187*	0.147 (7)
H17F	0.5734	−0.0102	0.4221	0.187*	0.147 (7)
C21	0.3059 (3)	0.5620 (2)	0.27442 (15)	0.0534 (6)	
C22	0.1754 (3)	0.6046 (3)	0.25292 (16)	0.0587 (6)	
H22	0.1232	0.6775	0.2803	0.070*	
C23	0.1162 (2)	0.5474 (3)	0.19387 (16)	0.0540 (6)	
C24	−0.0296 (3)	0.6042 (3)	0.1747 (2)	0.0749 (8)	
C25	−0.1095 (5)	0.4912 (5)	0.1775 (4)	0.124 (2)	0.846 (7)
H25A	−0.0608	0.4213	0.1424	0.186*	0.846 (7)
H25B	−0.1975	0.5216	0.1602	0.186*	0.846 (7)
H25C	−0.1203	0.4599	0.2316	0.186*	0.846 (7)
C26	−0.1031 (6)	0.7154 (8)	0.2291 (5)	0.171 (4)	0.846 (7)
H26A	−0.0513	0.7847	0.2265	0.256*	0.846 (7)
H26B	−0.1140	0.6852	0.2834	0.256*	0.846 (7)

H26C	−0.1910	0.7472	0.2121	0.256*	0.846 (7)
C27	−0.0206 (5)	0.6481 (6)	0.0866 (3)	0.112 (2)	0.846 (7)
H27A	0.0272	0.5766	0.0524	0.168*	0.846 (7)
H27B	0.0275	0.7201	0.0808	0.168*	0.846 (7)
H27C	−0.1106	0.6749	0.0715	0.168*	0.846 (7)
C25A	−0.1224 (17)	0.602 (3)	0.2530 (9)	0.100 (9)	0.154 (7)
H25D	−0.0859	0.6418	0.2953	0.149*	0.154 (7)
H25E	−0.1284	0.5132	0.2664	0.149*	0.154 (7)
H25F	−0.2115	0.6500	0.2466	0.149*	0.154 (7)
C26A	−0.024 (2)	0.7462 (13)	0.1521 (18)	0.103 (10)	0.154 (7)
H26D	0.0126	0.7878	0.1937	0.155*	0.154 (7)
H26E	−0.1138	0.7922	0.1461	0.155*	0.154 (7)
H26F	0.0335	0.7478	0.1022	0.155*	0.154 (7)
C27A	−0.088 (3)	0.537 (3)	0.1105 (14)	0.174 (18)	0.154 (7)
H27D	−0.0313	0.5382	0.0602	0.261*	0.154 (7)
H27E	−0.1781	0.5826	0.1049	0.261*	0.154 (7)
H27F	−0.0906	0.4484	0.1255	0.261*	0.154 (7)
C28	0.3632 (3)	0.6349 (3)	0.33910 (17)	0.0654 (7)	
C29	0.5132 (4)	0.6332 (4)	0.3144 (3)	0.1026 (13)	
H29A	0.5242	0.6805	0.2653	0.154*	
H29B	0.5598	0.5443	0.3061	0.154*	
H29C	0.5505	0.6736	0.3560	0.154*	
C210	0.2912 (4)	0.7776 (4)	0.3525 (3)	0.1086 (14)	
H21A	0.1959	0.7802	0.3685	0.163*	
H21B	0.3028	0.8248	0.3035	0.163*	
H21C	0.3298	0.8170	0.3941	0.163*	
C211	0.3453 (5)	0.5590 (4)	0.4167 (2)	0.1189 (16)	
H21D	0.2502	0.5603	0.4323	0.178*	
H21E	0.3828	0.5989	0.4585	0.178*	
H21F	0.3917	0.4702	0.4081	0.178*	
C31	0.1742 (3)	0.1101 (3)	0.1235 (2)	0.0767 (9)	
H31	0.1214	0.1172	0.1732	0.092*	
C32	0.1474 (4)	0.0242 (3)	0.0637 (3)	0.1010 (14)	
H32	0.0774	−0.0234	0.0737	0.121*	
C33	0.2274 (5)	0.0131 (4)	−0.0090 (3)	0.1017 (14)	
H33	0.2124	−0.0437	−0.0484	0.122*	
C34	0.3290 (4)	0.0847 (3)	−0.0242 (2)	0.0793 (10)	
C35	0.4177 (5)	0.0771 (4)	−0.0982 (2)	0.0993 (15)	
H35	0.4077	0.0204	−0.1390	0.119*	
C36	0.5151 (5)	0.1505 (4)	−0.1092 (2)	0.1042 (15)	
H36	0.5706	0.1435	−0.1580	0.125*	
C37	0.5365 (4)	0.2388 (3)	−0.04893 (16)	0.0776 (10)	
C38	0.6382 (4)	0.3153 (4)	−0.0552 (2)	0.0914 (12)	
H38	0.6952	0.3150	−0.1031	0.110*	
C39	0.6542 (4)	0.3892 (4)	0.0073 (2)	0.0899 (11)	
H39	0.7234	0.4383	0.0037	0.108*	
C40	0.5653 (3)	0.3912 (3)	0.07798 (19)	0.0673 (7)	
H40	0.5772	0.4428	0.1210	0.081*	
C41	0.4507 (3)	0.2462 (3)	0.02516 (14)	0.0573 (7)	

C42	0.3466 (3)	0.1692 (3)	0.03745 (15)	0.0587 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni	0.04364 (17)	0.05020 (19)	0.04083 (17)	−0.00254 (12)	−0.00597 (11)	−0.00931 (12)
O11	0.0509 (9)	0.0640 (11)	0.0451 (9)	0.0042 (8)	−0.0091 (7)	−0.0107 (8)
O12	0.0456 (9)	0.0842 (13)	0.0509 (10)	−0.0075 (9)	−0.0059 (8)	0.0050 (9)
O21	0.0495 (9)	0.0628 (11)	0.0581 (10)	−0.0014 (8)	−0.0083 (8)	−0.0225 (9)
O22	0.0506 (9)	0.0561 (10)	0.0542 (10)	0.0022 (8)	−0.0126 (8)	−0.0091 (8)
N31	0.0580 (12)	0.0483 (12)	0.0629 (13)	−0.0033 (10)	−0.0217 (10)	−0.0053 (10)
N32	0.0549 (12)	0.0519 (11)	0.0459 (11)	0.0018 (10)	−0.0038 (9)	−0.0046 (9)
C11	0.0517 (13)	0.0476 (13)	0.0486 (13)	−0.0042 (10)	−0.0127 (11)	−0.0071 (10)
C12	0.0579 (15)	0.0799 (19)	0.0461 (14)	−0.0070 (14)	−0.0107 (12)	0.0061 (13)
C13	0.0569 (15)	0.0591 (15)	0.0511 (14)	−0.0116 (12)	−0.0026 (11)	−0.0006 (12)
C14	0.0606 (17)	0.086 (2)	0.0623 (17)	−0.0110 (15)	0.0045 (13)	0.0150 (15)
C19	0.089 (3)	0.110 (3)	0.105 (3)	0.016 (2)	0.041 (2)	0.015 (2)
C110	0.095 (3)	0.324 (8)	0.054 (2)	−0.028 (4)	0.015 (2)	0.008 (3)
C111	0.101 (3)	0.097 (3)	0.198 (5)	−0.025 (3)	0.046 (3)	0.018 (3)
C18	0.0557 (15)	0.082 (2)	0.0572 (16)	−0.0018 (14)	−0.0208 (12)	−0.0065 (14)
C15	0.054 (2)	0.255 (9)	0.098 (4)	−0.041 (4)	−0.021 (2)	0.029 (5)
C16	0.079 (3)	0.182 (6)	0.102 (4)	0.014 (3)	−0.045 (3)	−0.075 (4)
C17	0.108 (4)	0.092 (3)	0.144 (5)	0.032 (3)	−0.054 (4)	−0.012 (3)
C15A	0.055 (11)	0.101 (15)	0.113 (16)	−0.003 (11)	−0.014 (11)	0.011 (13)
C16A	0.053 (11)	0.084 (14)	0.148 (19)	−0.007 (9)	−0.055 (12)	0.013 (13)
C17A	0.113 (14)	0.142 (15)	0.121 (14)	−0.010 (9)	−0.041 (9)	0.012 (10)
C21	0.0569 (14)	0.0541 (14)	0.0478 (13)	−0.0118 (12)	0.0043 (11)	−0.0123 (11)
C22	0.0529 (14)	0.0543 (15)	0.0629 (16)	0.0022 (11)	0.0009 (12)	−0.0140 (12)
C23	0.0478 (13)	0.0547 (15)	0.0558 (14)	−0.0023 (11)	−0.0012 (11)	0.0009 (12)
C24	0.0518 (16)	0.079 (2)	0.088 (2)	0.0073 (14)	−0.0120 (15)	−0.0029 (17)
C25	0.059 (3)	0.138 (5)	0.184 (7)	−0.029 (3)	−0.035 (3)	0.019 (4)
C26	0.087 (4)	0.182 (7)	0.215 (8)	0.075 (4)	−0.049 (5)	−0.112 (6)
C27	0.081 (3)	0.133 (5)	0.119 (4)	0.006 (3)	−0.039 (3)	0.030 (4)
C25A	0.034 (9)	0.132 (17)	0.114 (15)	0.027 (11)	0.002 (9)	0.030 (14)
C26A	0.057 (11)	0.116 (16)	0.132 (18)	0.005 (10)	−0.023 (11)	0.065 (14)
C27A	0.17 (2)	0.18 (2)	0.18 (2)	−0.001 (17)	−0.061 (18)	−0.010 (18)
C28	0.0764 (19)	0.0622 (17)	0.0590 (16)	−0.0174 (14)	−0.0045 (14)	−0.0214 (13)
C29	0.084 (2)	0.107 (3)	0.125 (3)	−0.033 (2)	−0.021 (2)	−0.043 (2)
C210	0.122 (3)	0.078 (2)	0.125 (3)	−0.006 (2)	−0.026 (3)	−0.053 (2)
C211	0.197 (5)	0.114 (3)	0.057 (2)	−0.052 (3)	−0.021 (3)	−0.021 (2)
C31	0.074 (2)	0.0637 (18)	0.099 (2)	−0.0155 (16)	−0.0317 (18)	−0.0010 (17)
C32	0.101 (3)	0.067 (2)	0.152 (4)	−0.024 (2)	−0.070 (3)	0.000 (2)
C33	0.131 (4)	0.068 (2)	0.109 (3)	0.010 (2)	−0.073 (3)	−0.024 (2)
C34	0.103 (2)	0.0591 (17)	0.075 (2)	0.0149 (17)	−0.0519 (19)	−0.0184 (15)
C35	0.136 (4)	0.092 (3)	0.0541 (19)	0.043 (2)	−0.047 (2)	−0.0327 (18)
C36	0.131 (4)	0.115 (3)	0.0415 (16)	0.048 (3)	−0.016 (2)	−0.0148 (19)
C37	0.095 (2)	0.077 (2)	0.0420 (14)	0.0336 (18)	−0.0055 (14)	−0.0010 (14)
C38	0.084 (2)	0.102 (3)	0.070 (2)	0.014 (2)	0.0207 (18)	0.022 (2)

C39	0.075 (2)	0.088 (2)	0.096 (3)	−0.0022 (18)	0.0172 (19)	0.017 (2)
C40	0.0575 (16)	0.0669 (18)	0.0730 (19)	−0.0047 (14)	0.0008 (14)	0.0042 (14)
C41	0.0665 (16)	0.0555 (15)	0.0408 (13)	0.0153 (13)	−0.0096 (11)	−0.0011 (11)
C42	0.0735 (17)	0.0507 (14)	0.0461 (14)	0.0158 (13)	−0.0253 (12)	−0.0097 (11)

Geometric parameters (Å, °)

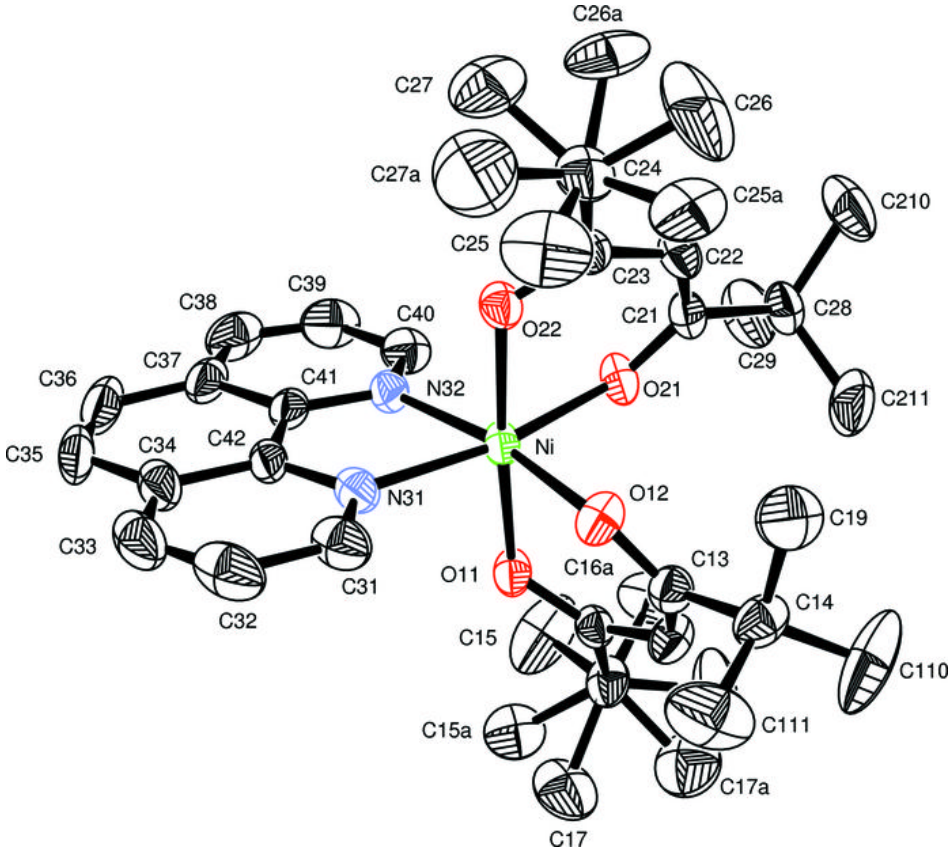
Ni—O12	2.0110 (18)	C22—H22	0.9300
Ni—O21	2.0176 (18)	C23—C24	1.547 (4)
Ni—O11	2.0304 (18)	C24—C26	1.502 (5)
Ni—O22	2.0320 (18)	C24—C27A	1.508 (8)
Ni—N32	2.089 (2)	C24—C25A	1.520 (8)
Ni—N31	2.094 (2)	C24—C26A	1.531 (8)
O11—C11	1.269 (3)	C24—C25	1.535 (5)
O12—C13	1.266 (3)	C24—C27	1.537 (5)
O21—C21	1.268 (3)	C25—H25A	0.9600
O22—C23	1.262 (3)	C25—H25B	0.9600
N31—C31	1.312 (4)	C25—H25C	0.9600
N31—C42	1.358 (3)	C26—H26A	0.9600
N32—C40	1.323 (4)	C26—H26B	0.9600
N32—C41	1.350 (3)	C26—H26C	0.9600
C11—C12	1.395 (4)	C27—H27A	0.9600
C11—C18	1.539 (3)	C27—H27B	0.9600
C12—C13	1.394 (4)	C27—H27C	0.9600
C12—H12	0.9300	C25A—H25D	0.9600
C13—C14	1.537 (4)	C25A—H25E	0.9600
C14—C19	1.505 (4)	C25A—H25F	0.9600
C14—C111	1.528 (5)	C26A—H26D	0.9600
C14—C110	1.532 (5)	C26A—H26E	0.9600
C19—H19A	0.9600	C26A—H26F	0.9600
C19—H19B	0.9600	C27A—H27D	0.9600
C19—H19C	0.9600	C27A—H27E	0.9600
C110—H11A	0.9600	C27A—H27F	0.9600
C110—H11B	0.9600	C28—C29	1.515 (5)
C110—H11C	0.9600	C28—C211	1.523 (5)
C111—H11D	0.9600	C28—C210	1.533 (4)
C111—H11E	0.9600	C29—H29A	0.9600
C111—H11F	0.9600	C29—H29B	0.9600
C18—C16	1.501 (4)	C29—H29C	0.9600
C18—C15A	1.508 (9)	C210—H21A	0.9600
C18—C15	1.513 (5)	C210—H21B	0.9600
C18—C17A	1.524 (9)	C210—H21C	0.9600
C18—C16A	1.531 (9)	C211—H21D	0.9600
C18—C17	1.541 (5)	C211—H21E	0.9600
C15—H15A	0.9600	C211—H21F	0.9600
C15—H15B	0.9600	C31—C32	1.423 (5)
C15—H15C	0.9600	C31—H31	0.9300
C16—H16A	0.9600	C32—C33	1.373 (6)
C16—H16B	0.9600	C32—H32	0.9300

C16—H16C	0.9600	C33—C34	1.368 (6)
C17—H17A	0.9600	C33—H33	0.9300
C17—H17B	0.9600	C34—C42	1.405 (4)
C17—H17C	0.9600	C34—C35	1.433 (6)
C15A—H15D	0.9600	C35—C36	1.341 (6)
C15A—H15E	0.9600	C35—H35	0.9300
C15A—H15F	0.9600	C36—C37	1.427 (5)
C16A—H16D	0.9600	C36—H36	0.9300
C16A—H16E	0.9600	C37—C38	1.398 (5)
C16A—H16F	0.9600	C37—C41	1.421 (4)
C17A—H17D	0.9600	C38—C39	1.341 (5)
C17A—H17E	0.9600	C38—H38	0.9300
C17A—H17F	0.9600	C39—C40	1.395 (4)
C21—C22	1.393 (4)	C39—H39	0.9300
C21—C28	1.543 (3)	C40—H40	0.9300
C22—C23	1.396 (4)	C41—C42	1.424 (4)
O12—Ni—O21	95.30 (8)	C26—C24—C25A	50.1 (9)
O12—Ni—O11	88.56 (7)	C27A—C24—C25A	107.9 (9)
O21—Ni—O11	89.12 (7)	C26—C24—C26A	59.3 (9)
O12—Ni—O22	89.61 (8)	C27A—C24—C26A	110.4 (9)
O21—Ni—O22	87.79 (7)	C25A—C24—C26A	109.3 (8)
O11—Ni—O22	176.25 (6)	C26—C24—C25	110.9 (5)
O12—Ni—N32	170.45 (8)	C27A—C24—C25	47.6 (12)
O21—Ni—N32	92.88 (8)	C25A—C24—C25	67.2 (10)
O11—Ni—N32	86.63 (8)	C26A—C24—C25	147.6 (8)
O22—Ni—N32	95.65 (8)	C26—C24—C27	110.2 (4)
O12—Ni—N31	93.42 (9)	C27A—C24—C27	57.6 (11)
O21—Ni—N31	170.00 (8)	C25A—C24—C27	145.5 (8)
O11—Ni—N31	95.99 (8)	C26A—C24—C27	58.3 (11)
O22—Ni—N31	87.38 (8)	C25—C24—C27	105.1 (4)
N32—Ni—N31	78.89 (9)	C26—C24—C23	114.6 (3)
C11—O11—Ni	121.76 (15)	C27A—C24—C23	118.4 (14)
C13—O12—Ni	124.30 (17)	C25A—C24—C23	106.4 (8)
C21—O21—Ni	124.38 (16)	C26A—C24—C23	104.2 (8)
C23—O22—Ni	122.52 (16)	C25—C24—C23	107.6 (3)
C31—N31—C42	118.4 (3)	C27—C24—C23	108.0 (3)
C31—N31—Ni	128.4 (2)	C24—C25—H25A	109.5
C42—N31—Ni	113.25 (18)	C24—C25—H25B	109.5
C40—N32—C41	117.9 (2)	H25A—C25—H25B	109.5
C40—N32—Ni	128.40 (19)	C24—C25—H25C	109.5
C41—N32—Ni	113.63 (19)	H25A—C25—H25C	109.5
O11—C11—C12	124.6 (2)	H25B—C25—H25C	109.5
O11—C11—C18	115.5 (2)	C24—C26—H26A	109.5
C12—C11—C18	119.9 (2)	C24—C26—H26B	109.5
C13—C12—C11	126.1 (2)	H26A—C26—H26B	109.5
C13—C12—H12	117.0	C24—C26—H26C	109.5
C11—C12—H12	117.0	H26A—C26—H26C	109.5
O12—C13—C12	124.4 (2)	H26B—C26—H26C	109.5
O12—C13—C14	115.1 (2)	C24—C27—H27A	109.5

C12—C13—C14	120.6 (2)	C24—C27—H27B	109.5
C19—C14—C111	108.5 (3)	H27A—C27—H27B	109.5
C19—C14—C110	107.9 (4)	C24—C27—H27C	109.5
C111—C14—C110	110.4 (4)	H27A—C27—H27C	109.5
C19—C14—C13	109.9 (2)	H27B—C27—H27C	109.5
C111—C14—C13	106.7 (3)	C24—C25A—H25D	109.5
C110—C14—C13	113.4 (3)	C24—C25A—H25E	109.5
C14—C19—H19A	109.5	H25D—C25A—H25E	109.5
C14—C19—H19B	109.5	C24—C25A—H25F	109.5
H19A—C19—H19B	109.5	H25D—C25A—H25F	109.5
C14—C19—H19C	109.5	H25E—C25A—H25F	109.5
H19A—C19—H19C	109.5	C24—C26A—H26D	109.5
H19B—C19—H19C	109.5	C24—C26A—H26E	109.5
C14—C110—H11A	109.5	H26D—C26A—H26E	109.5
C14—C110—H11B	109.5	C24—C26A—H26F	109.5
H11A—C110—H11B	109.5	H26D—C26A—H26F	109.5
C14—C110—H11C	109.5	H26E—C26A—H26F	109.5
H11A—C110—H11C	109.5	C24—C27A—H27D	109.5
H11B—C110—H11C	109.5	C24—C27A—H27E	109.5
C14—C111—H11D	109.5	H27D—C27A—H27E	109.5
C14—C111—H11E	109.5	C24—C27A—H27F	109.5
H11D—C111—H11E	109.5	H27D—C27A—H27F	109.5
C14—C111—H11F	109.5	H27E—C27A—H27F	109.5
H11D—C111—H11F	109.5	C29—C28—C211	109.6 (3)
H11E—C111—H11F	109.5	C29—C28—C210	108.5 (3)
C16—C18—C15A	131.7 (11)	C211—C28—C210	109.8 (3)
C16—C18—C15	112.7 (4)	C29—C28—C21	109.2 (2)
C15A—C18—C15	58.8 (12)	C211—C28—C21	106.3 (2)
C16—C18—C17A	44.4 (16)	C210—C28—C21	113.4 (3)
C15A—C18—C17A	104.9 (19)	C28—C29—H29A	109.5
C15—C18—C17A	135.8 (14)	C28—C29—H29B	109.5
C16—C18—C16A	62.1 (12)	H29A—C29—H29B	109.5
C15A—C18—C16A	118.1 (16)	C28—C29—H29C	109.5
C15—C18—C16A	61.6 (13)	H29A—C29—H29C	109.5
C17A—C18—C16A	106 (2)	H29B—C29—H29C	109.5
C16—C18—C11	113.2 (3)	C28—C210—H21A	109.5
C15A—C18—C11	113.8 (10)	C28—C210—H21B	109.5
C15—C18—C11	110.8 (3)	H21A—C210—H21B	109.5
C17A—C18—C11	113.2 (14)	C28—C210—H21C	109.5
C16A—C18—C11	101.1 (7)	H21A—C210—H21C	109.5
C16—C18—C17	108.1 (4)	H21B—C210—H21C	109.5
C15A—C18—C17	46.9 (12)	C28—C211—H21D	109.5
C15—C18—C17	104.9 (4)	C28—C211—H21E	109.5
C17A—C18—C17	65.7 (17)	H21D—C211—H21E	109.5
C16A—C18—C17	152.2 (8)	C28—C211—H21F	109.5
C11—C18—C17	106.6 (3)	H21D—C211—H21F	109.5
C18—C15—H15A	109.5	H21E—C211—H21F	109.5
C18—C15—H15B	109.5	N31—C31—C32	122.0 (4)
C18—C15—H15C	109.5	N31—C31—H31	119.0

C18—C16—H16A	109.5	C32—C31—H31	119.0
C18—C16—H16B	109.5	C33—C32—C31	118.4 (4)
C18—C16—H16C	109.5	C33—C32—H32	120.8
C18—C17—H17A	109.5	C31—C32—H32	120.8
C18—C17—H17B	109.5	C34—C33—C32	120.9 (3)
C18—C17—H17C	109.5	C34—C33—H33	119.6
C18—C15A—H15D	109.5	C32—C33—H33	119.6
C18—C15A—H15E	109.5	C33—C34—C42	117.0 (4)
H15D—C15A—H15E	109.5	C33—C34—C35	124.0 (3)
C18—C15A—H15F	109.5	C42—C34—C35	118.9 (4)
H15D—C15A—H15F	109.5	C36—C35—C34	121.0 (3)
H15E—C15A—H15F	109.5	C36—C35—H35	119.5
C18—C16A—H16D	109.5	C34—C35—H35	119.5
C18—C16A—H16E	109.5	C35—C36—C37	122.4 (4)
H16D—C16A—H16E	109.5	C35—C36—H36	118.8
C18—C16A—H16F	109.5	C37—C36—H36	118.8
H16D—C16A—H16F	109.5	C38—C37—C41	117.0 (3)
H16E—C16A—H16F	109.5	C38—C37—C36	125.6 (4)
C18—C17A—H17D	109.5	C41—C37—C36	117.4 (4)
C18—C17A—H17E	109.5	C39—C38—C37	120.5 (3)
H17D—C17A—H17E	109.5	C39—C38—H38	119.7
C18—C17A—H17F	109.5	C37—C38—H38	119.7
H17D—C17A—H17F	109.5	C38—C39—C40	118.9 (4)
H17E—C17A—H17F	109.5	C38—C39—H39	120.6
O21—C21—C22	124.3 (2)	C40—C39—H39	120.6
O21—C21—C28	114.1 (2)	N32—C40—C39	123.5 (3)
C22—C21—C28	121.6 (2)	N32—C40—H40	118.2
C21—C22—C23	125.7 (2)	C39—C40—H40	118.2
C21—C22—H22	117.1	N32—C41—C37	122.1 (3)
C23—C22—H22	117.1	N32—C41—C42	117.1 (2)
O22—C23—C22	124.6 (2)	C37—C41—C42	120.7 (3)
O22—C23—C24	114.3 (2)	N31—C42—C34	123.3 (3)
C22—C23—C24	121.1 (2)	N31—C42—C41	117.1 (2)
C26—C24—C27A	126.8 (14)	C34—C42—C41	119.6 (3)

Fig. 1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